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ECOL-002GB

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0223792.3 — 14 OCT 2002

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Dr. Boris Zachar Gorbunov
20 Kings Park
Canterbury
Kent CT1 1QH

Patents ADP number *(if you know it)*

7103310002

If the applicant is a corporate body, give the country/state of its incorporation

4. Title of the invention

The Method of and Apparatus for Counting Ions in a Sample

5. Name of your agent *(if you have one)*

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

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Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

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a) any applicant named in part 3 is not an inventor, or
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Continuation sheets of this form	1
Description	7
Claim *(s)*	3
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Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	
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11. I/We request the grant of a patent on the basis of this application.

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Date 11/10/02

12. Name and daytime telephone number of person to contact in the United Kingdom: B. GORBUNOV 01227-787-589

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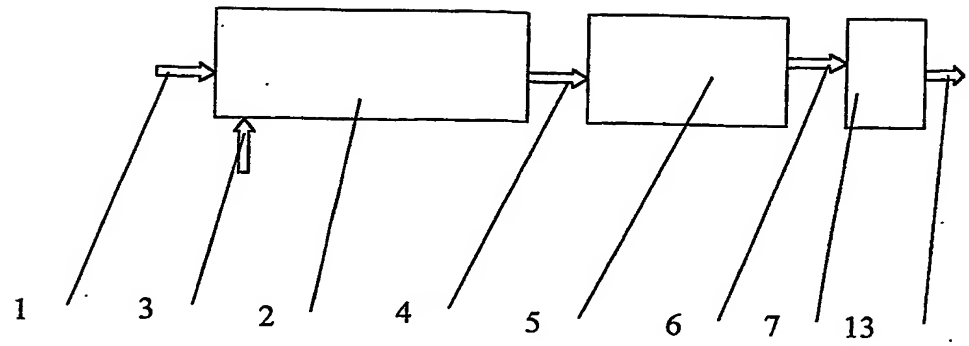

Fig. 1.

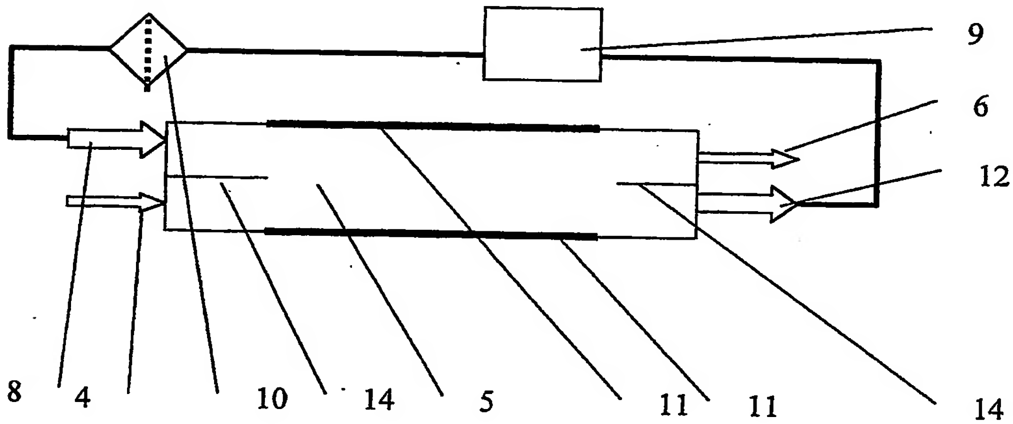

Fig. 2.

This invention relates to method of and apparatus for counting ions in a sample. More particularly, the invention relates to quantifying extremely low concentrations of ions in a gaseous medium.

In the book titled "Plasma chromatography" Edited by T.W. Carr and published in 1984 by Plenum Press (N-Y, London) there is described the measurement of concentrations of ions and molecules in a gas medium by means of electric current detection with an electrometer or other current detecting device. In a conventional electrical detector, based upon the Faraday cup, ions impinge on the collector and carry an electric charge. The voltage drop across the standard resistor connected to the collector is a measure of the ion current. The voltage signal from the resistor is then amplified by an amplifier. The measured and amplified ion currents are directly proportional to the number of ions and number of charges per ion. Therefore, the response of the Faraday cup depends upon the number of the ions collided with the collector. Faraday cup detectors are simple, inexpensive, rugged and reliable. They have high accuracy and constant sensitivity. The principal disadvantage of the Faraday cup is that the low detection limit of ions is relatively high. It is caused by its amplification system and electrical noise. Thus, this prior art method cannot be used to quantify extremely low concentrations of ions in gases.

It is an object of the present invention to provide a method for quantifying concentration of ions in gases avoiding the use of a Faraday cup device or other devices with collecting electrodes and measuring electric current.

Accordingly, one aspect of the present invention provides a method of counting ions in a sample including colliding the ions with uncharged particles of greater mass than the ions to transfer charge from respective ions to individual particles and selecting particles charged following collision by subjecting the sample to an electric field to direct the charged particles toward means arranged to detect and numerically count the charged particles.

Preferably, the number concentration of the uncharged particles is greatly in excess of the number concentration of the ions.

Suitably, the charged particles are detected individually by means of single particle counting means.

Another aspect of the present invention provides apparatus for counting ions in a sample including means arranged to promote a flow of the sample in a gaseous form into a mixing chamber, means arranged to promote a flow of uncharged aerosol particles entrained in an air flow into the mixing chamber to collide with the ions, an outlet from the mixing chamber discharging to a separation chamber provided with electrode means arranged to subject the separation chamber to an electric field and an outlet from the separating chamber discharging to a particle detecting and numerically measuring means.

Desirably, evaporator and condensation means are provided to produce the uncharged aerosol particles.

The invention will now be described, by way of example, with reference to the accompanying schematic drawings in which:
FIGURE 1 is apparatus for detecting the presence of, and measuring extremely low concentrations of ions in gases; and
FIGURE 2 is a view of a separation chamber together with some associated equipment.

Referring to fig. 1, there is shown a mixing chamber 2 having a first inlet 1 for a sample gas flow containing ions, a second inlet 3 for uncharged aerosol particles entrained in a flow of air and an outlet 4 discharging to a separation chamber 5. The separation chamber is provided with electrodes 11, producing an electric field, and discharges through a connector 6 to optical particle counter 7.

In operation, sample gas containing ions enters the mixing chamber 2 through

the first inlet 1 with the flow of the gas sample effected either by force flow maintaining means (not shown) at the inlet or induced flow maintaining means (not shown) at the exhaust outlet 13 from the apparatus. In the mixing chamber 2, the sample gas flow containing the ions is mixed with the air flow carrying the uncharged aerosol particles introduced into the mixing chamber 2 through the inlet 3 with the concentration of the uncharged aerosol particles greatly in excess of the concentration of ions. The ions and aerosol particles collide with each other as a result of Brownian diffusion and some of the aerosol particles become charged by transfer of charge from the ions. Thus, the flow discharged from the mixing chamber 2 contains both charged and uncharged aerosol particles and passes to the separation chamber 5, where charged and uncharged aerosol particles are separated according to their electric mobility by the imposition of the electric field to the effect that at the outlet 6 from the chamber 5 the gas flow contains only charged aerosol particles.

The charged aerosol particles entrained in the gas flow discharge through connector 6 into the optical particle counter 7 where the charged aerosol particles are detected and counted. Since the charged aerosol particles have acquired the electric charges following collisions with ions, the number of charged aerosol particles is substantially related to the number of ions in the mixing chamber 2. For unit flow rates, the number of ions in the mixing chamber 2 is proportionate to the number of ions entered the mixing chamber 2 in the sample gas so that the concentration of charged particles is indicative of the concentration of ions in the sample gas.

A correction factor links the actual concentration of ions with the number concentration of charged particles is found by means of calibration using mass spectrometry or another suitable techniques.

In one arrangement of the separation chamber 5 (which may be in the form of a commercially available differential mobility analyser column) as shown in Figure 2, following separation, the gases with entrained neutral particles are

recycled through an outlet 12, a pump means 9 and an aerosol fibre filter means 10 to an inlet 8. The electrodes 11 are positioned at upper and lower regions of the chamber 5, the inlet 8 and the outlet 6 are respectively positions at the upper region of the chamber, whilst the inlet 4 and outlet 12 are respectively positioned at the lower region. Flow dividing baffles 14 are positioned in end regions of the chamber.

In operation, the gas flow with both charged and neutral aerosol particles enters the separation chamber 5 through the inlet 4 and the neutral particles are carried with the gas flow to the outlet 12. The charged particles are urged upwardly and towards the outlet 6 by the effect of the electric field generated by the energised electrodes 11 thereby urging the charged particles into the flow of filtered gas from the inlet 8. The flow rates of the gases in the separation chamber 5 have to satisfy "the laminar flow criterion": the linear velocity of the flows have to be equal.

In the embodiment of the apparatus, the mixing chamber 2 was manufactured from brass having the shape of a cylinder of the internal volume of 0.5 litres. All the inlets and connectors were made from brass and cooper. The separation chamber 5 was of rectangular cross-section and manufactured from aluminium with cooper electrodes 11 insulated and placed inside the chamber. The distance between the electrodes was 5 mm and the voltage was from 1000 to 10,000 Volts DC.

A commercial optical aerosol particle counter MetOne (Pacific Scientific Instruments) was used to count particles. This optical particle counter enables aerosol particles of the diameter greater than 0.3 μm to be individually counted.

In a test ions have been formed in the air using ^{241}Am (0.9 μCi) α -particle emitter. Uncharged aerosol particles were generated from glycerol by an aerosol generator based upon gas-to-particle conversion mechanism. The particle number concentration depends upon the flow rate through the aerosol

generator and evaporation temperature. The number concentration of glycerol particles was in the range from 10^9 to 10^{12} m^{-3}. The flow rate was maintained by a pump and quantified by a rotameter: the range of the flow rate was from 0.2 to 2 liters/min.

The sample air flow containing the ions was drawn to the mixing chamber 2 through the inlet 1 by an exhaust pump (not shown) connected to the optical particle counter 7 to mix with the uncharged glycerol aerosol particle flow in the mixing chamber. In the mixing chamber 2 the ions collided with the uncharged glycerol aerosol particles and some of aerosol particles became charged. The flow discharged from the mixing chamber 2 contained both charged and uncharged glycerol particles. The flow containing the charged and uncharged aerosol particles than entered the separation chamber 5 through the outlet connector 4. In the separation chamber 5, charged and uncharged glycerol aerosol particles were separated according to their electric mobility in the electric field such that at the outlet 6 of the chamber 5, the gas flow contained only charged glycerol aerosol particles of about 1 μm mean diameter. The charged glycerol aerosol particles were carried by the gas flow through the outlet connector 6 into the optical particle counter 7. The concentration of ions in the air formed by the radioactive source ^{241}Am was found to be 2×10^2 cm^{-3}. In other experiments, recorded concentrations were in the range from 7 to 3000 cm^{-3}.

It will be appreciated that any type of semi-volatile material can be used to generate aerosol particles, for instance sulphur. These aerosol particles suspended in a gas may be either liquid or solid. Aerosol particles may be produced from a mixture of organic compounds or inorganic substances. Atmospheric aerosol particles may also be used to accept charges in the mixing chamber. Aerosols particles may be also generated from a dust or from a liquid using an atomiser as well as nebuliser. Particles also may be in a liquid in the form of a hydrosol or emulsion. Aerosol particles generated in these ways have charges. These charges have to be removed by means employed to remove charged particles from the aerosol flow.

It will also be appreciated that an ion mobility selection unit may be attached to the inlet 1 to enable ions of pre-determined mobility to pass into the mixing chamber 2. Thus, the ions with such pre-determined mobility are selected for the detection and measurement.

It will further be appreciated that an ionisation chamber containing means for effecting ionisation of molecules or clusters of interest, for instance UV radiation of about 10 or 11 eV may be attached to the inlet 1 of the ion counter. This enables a wide range of species, e.g. molecules, free radicals, clusters, nano-particles, and atoms, to be detected and quantified.

For detection of trace species in liquids or solids a liquid or a solid sample may be evaporated first into a gas medium and then treated as a gas sample. Alternatively, a liquid or solid sample may be heated to a predetermined temperature first to release some of the trace species in a gas medium and then the gas medium containing the trace species may be treated as a gas sample.

It will also further be appreciated that the ionisation means may comprise a method of ionisation with a degree of selectivity. In the case of photo-ionisation, the ionisation selectivity may be achieved by choosing the gas containing molecules or atoms with a higher ionisation potential than the energy of the UV source.

Counting aerosol particles enables very low concentrations of ionised matter to be quantified on-line, e.g. concentrations as low as 1 cm^{-3} may be measured reliably. Conventional equipment to count ions in a gas is reliable only down to concentrations of 10^5 cm^{-3}.

Where necessary, a plurality of mixing chambers, arranged in series or in parallel, may be provided. Furthermore, where necessary, a plurality of selection chambers, or particle generator means, arranged in series or in parallel, may be provided.

Other particle counting means may be utilised such as light scattering or light absorption detectors or a dust monitor, nephelometer, aethelometer or a condensation particle counter.

Where advantageous, other detectable species such as clusters, nano-particles, molecules suspended in a gas may be used instead of uncharged aerosol particles.

If desired, a condensation unit, adapted to increase the size and the mass of the charged aerosol particles or the detectable species may be positioned in intermediate the separation chamber 5 and the charged aerosol particle detector.

Where appropriate, charge neutralisation or charge removal means may be positioned in flows at the inlet 3 and 8 containing uncharged aerosol particles to ensure the neutrality of such flows.

If desired, a differential mobility analyser may serve as separation chamber 5 providing a plurality of outputs according to particle mobility.

CLAIMS

1. A method of counting ions in a sample including colliding the ions with uncharged particles of greater mass than the ions to transfer charge from respective ions to individual particles and selecting particles charged following collision by subjecting the sample to an electric field to direct the charged particles toward means arranged to detect and numerically count the charged particles.

2. A method of counting ions in a sample as claimed in Claim 1, wherein the number concentration of the uncharged particles is greatly in excess of the number concentration of the ions.

3. A method of counting ions in a sample as claimed in Claim 1 or Claim 2, wherein the sample is a steady flow of gas containing ions and is combined and mixed with a steady flow of uncharged aerosol particles, or nano-particles, or clusters, or molecules entrained in a gas and the combined flow is subjected to the electric field.

4. A method of counting ions in a sample as claimed in Claim 3, wherein the uncharged aerosol particles are produced by evaporating glycerol and condensing glycerol vapours in a steady flow of air.

5. A method of counting ions in a sample as claimed in Claim 3 or Claim 4, wherein the charged particles are increased in mass and size by subjecting the charged particles to a condensation process.

6. A method of counting ions in a sample as claimed in Claim 3, Claim 4 or Claim 5, wherein the gas flow is laminar in the vicinity of the electric field and the charged particles migrate to a location in the gas flow cross-section dependant upon the electric mobility of the charged particles and the strength of the electric field.

7. A method of counting ions in a sample as claimed in Claim 6, wherein the charged particles impinge upon the detecting and numeric counting means in a manner indicative of the magnitude of the respective charge.

8. A method of counting ions in a sample as claimed in any preceding claim, wherein the means arranged to detect and numerically count the charged particles are an optical particle counter adapted to count individual charged particles.

9. Apparatus for counting ions in a sample including means arranged to promote a flow of the sample in a gaseous form into a mixing chamber, means arranged to promote a flow of uncharged aerosol particles entrained in an air flow into the mixing chamber to collide with the ions, an outlet from the mixing chamber discharging to a separation chamber provided with electrode means arranged to subject the separation chamber to an electric field and an outlet from the separating chamber discharging to a particle detecting and numerically measuring means.

10. Apparatus for counting ions in a sample as claimed in Claim 9, wherein evaporator and, if appropriate, condensation means are arranged to produce the uncharged aerosol particles, or uncharged nano-particles, or neutral clusters, or molecules suspended in a gas medium.

11. Apparatus for counting ions in a sample as claimed in Claim 9 or Claim 10, wherein a further outlet from the separation chamber is connected through pump means and aerosol fibre filter means to a further inlet to the separating chamber discharging in parallel to and adjacent the inlet to the separating chamber from the mixing chamber or a differential mobility analyser is employed as a separation chamber.

12. Apparatus for counting ions in a sample as claimed in Claim 11, wherein short baffles extending in the direction of flow are positioned within the separation chamber respectively adjacent inlet and outlet regions.

13. Apparatus for counting ions in a sample as claimed in Claim 9 or Claim 10, wherein atmospheric aerosols first neutralised by means of neutralisation or charge removal means and than are utilised to accept charges from ions.

14. Apparatus for counting ions in a sample as claimed in any one of Claims 9 to13, wherein the particle detecting and numerically measuring means is an optical particle counter, or a condensation particle counter, or nephelometer.

15. Apparatus for counting ions in a sample arranged and adapted to operate substantially as hereinbefore described with reference to Figure 1 or with reference to Figures 1 and 2 of the accompanying drawings.

16. A method of counting ions in a sample substantially as hereinbefore described.

ABSTRACT

THE METHOD OF AND APPARATUS FOR COUNTING IONS IN A SAMPLE

Ions in a steady flow sample are counted (Figure 1) by colliding the ions in a mixing chamber 2 with a numerical excess of uncharged aerosol particles, suitably glycerol, entrained in air, to transfer respective charges from the ions to charge individual aerosol particles and passing the gases through a separating chamber 5 subjected to an electric field to direct the charged aerosol particles to impinge upon an optical particle counter 7.

(Figure 1 to accompany abstract)